Time dependence of energy dissipation in resonating silicon cantilevers in ultrahigh vacuum

doi: 10.1063/1.1608485

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(Received 7 April 2003; accepted 8 July 2003)

The nanomechanical properties of single-crystalline silicon cantilevers as resonating elements are investigated in ultra-high vacuum. Flash heating the single-crystalline silicon cantilever at 1000 °C in ultra-high vacuum dramatically reduced the mechanical energy dissipation of the oscillating cantilever due to the removal of contaminants and natural oxide and reconstruction of the silicon surface. However, the reconstructed silicon surface is not stable and mechanical properties change depending on the surface state. The Q-factor and influence of gas adsorption on ultrathin single crystalline silicon resonators having (100) and (111)-oriented surfaces are presented. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1608485]

Small resonators fabricated by micromachining have been widely used as force sensors in scanning probe microscopy (SPM), and can detect tiny physical interactions with a sample through van der Waals forces, electrostatic forces, magnetic forces, etc.[1–6] Many applications stimulate continuous efforts to improve the performance of these sensors toward the ultimate limit of sensing.[1–3]

Thermomechanical vibration of the oscillator is the dominant noise source under most conditions, and limits the detectable minimum force $F_{\min}$ according to[4]

$$F_{\min} = \sqrt{\frac{2kk_BTB}{\pi f_0 Q}}, \tag{1}$$

where k the spring constant, k_B the Boltzmann constant, T the temperature, Q the quality factor, and f_0 the resonant frequency. As can be seen from Eq. (1), smaller structures with high resonant frequency and small spring constants are expected to lead to smaller detectable forces and higher mechanical response as long as a high mechanical Q factor is maintained. Mechanical Q factors exhibit the energy dissipation of vibrating systems in which a high Q factor provides a low energy loss. This physical behavior, predicted by scaling effects, is the main driving force behind the miniaturization of resonating sensors. However, Q factors are decreasing as thickness decreases due to energy dissipation on the surface, the so-called ''surface loss,'' [3,5] due to friction from surface defects and adsorbates. Previous experiments show that the Q factor of very thin cantilever beams decreased due to adsorption of oxygen.[7] Unfortunately, the clean silicon surface is very reactive and is gradually oxidized, even in ultrahigh vacuum (UHV). For many applications, this instability of mechanical properties is a serious problem. In this letter, the mechanical properties are characterized, especially focusing on changes in mechanical properties due to adsorption under UHV conditions.

All measurements were performed in an UHV system equipped with a laser Doppler vibrometer under a pressure of 1×10^{-7} Pa at room temperature, and thermal treatments were performed in a treatment chamber under a pressure of 2×10^{-8} Pa. Fabrication of the resonators is described in Ref. 8. Fabricated resonators were mounted on a sample holder and introduced into the treatment chamber through a load–lock chamber and the cantilever was heated by passing a current through the silicon base supporting the cantilever. The dimensions, resonant frequencies and spring constants of measured cantilever are listed in Table I. Surface structures were confirmed by reflection high energy electron diffraction (RHEED), with the 2×1 reconstruction observed for Si(100) and the 7×7 reconstruction observed for Si(111). In addition, streaks of diffraction patterns instead of spots indicate a small roughness on the silicon surface in both cases. The sample was then introduced into the measurement chamber. The cantilever was actuated by applying a voltage

TABLE I. Dimensions of the measured cantilevers with rectangular shape.

Sample No.	Orientation	Width (μm)	Length (μm)	Thickness (nm)	Resonant frequency (kHz)	Spring constants (N/m)
1	Si (100)	7	58	170	69	7.5×10^{-3}
2	Si (100)	7	49	170	98	1.2×10^{-2}
3	Si (111)	7	33	50	64	1.0×10^{-2}
4	Si (111)	4	43	50	37	2.7×10^{-4}

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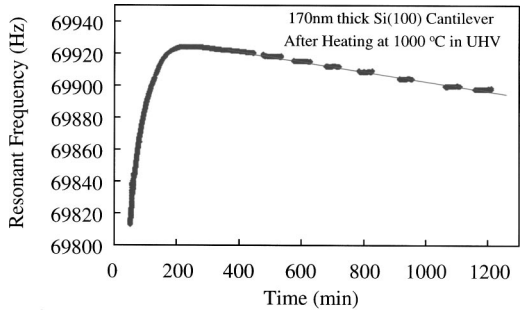


FIG. 1. Time dependence of the resonant frequency of 170 nm thick (100)-oriented Si cantilever (sample No. 1) after annealing in UHV.

between the cantilever and a metal electrode located ~6 mm above the cantilever. The laser beam of the Doppler vibrometer, which has a diameter of 1.5 μm and power of ~1 mW, was focused onto the cantilever to measure vibrations. The velocity signal from the Doppler vibrometer was fed to the metal electrode through an amplifier, filter and wave converter, self-oscillating the cantilever. The vibrational frequency was measured by a pulse counter (Advantest R5363) with an aging rate of 5×10^{-10}/day, and the amplitude was measured by a lockin amplifier. The Q factor was obtained from the step response of the oscillating cantilever. When the feed signal was abruptly turned off, the amplitude of the oscillating cantilever decayed at a time constant τ, from which the Q factor can be calculated according to $Q=\pi f_0\tau$.

The stability of mechanical properties is significant in most applications. The relationship between surface state and the mechanical properties of the cantilever may give an indication of sensitivity and long-term stability. Time dependence of resonant frequency and Q factor of the cantilever, which were flash heated to prepare a clean surface, were measured in UHV.

Figure 1 shows the resonant frequency of a (100)-oriented Si cantilever (sample No. 1) after being heated to 1000 °C. The cantilever was immediately transferred into the measurement chamber at a base pressure of 1×10^{-7} Pa. Time dependence of the Q factor of a (100) cantilever (sample No. 2) is shown in Fig. 2. Figure 3 shows the resonant frequency and Q factor of a (111)-oriented cantilever (sample No. 3).

From Fig. 1, the resonant frequency of the (100)-oriented cantilever can be seen to have increased up to ~200 min after heating and then gradually decreased. The resonant frequency of the silicon resonator is known to increase with decreasing temperature at a rate of 35 ppm/°C, and thus temperature change is the dominant factor in frequency changes in the early stages of heating. In contrast to the Si(100) cantilever, the resonant frequency of the (111)-oriented cantilever continues to drift higher in Fig. 3(a). A combination of mass increase due to molecular adsorption and surface stress induced by the adsorption determines the behavior of the resonant frequency. In UHV, the main residual gas molecules available for adsorption are water, which dissociate on the surface to a hydroxyl group and hydrogen atoms.

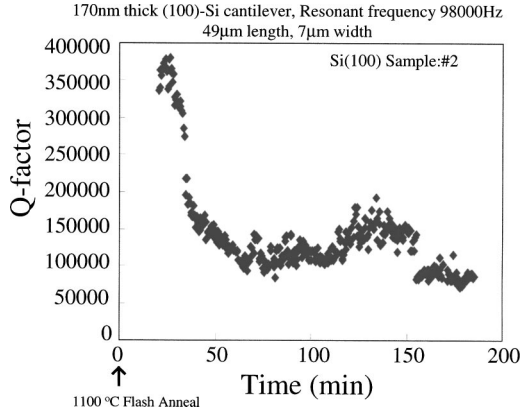
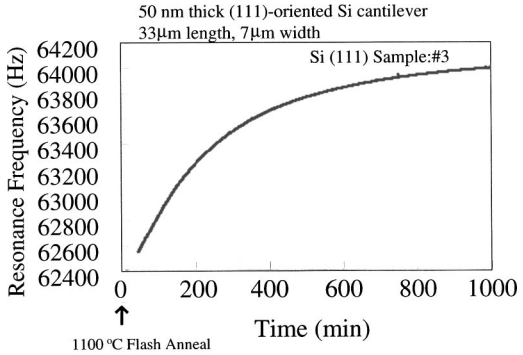


FIG. 2. Time dependence of Q factor of Si(100) cantilever (sample No. 2) after annealing.



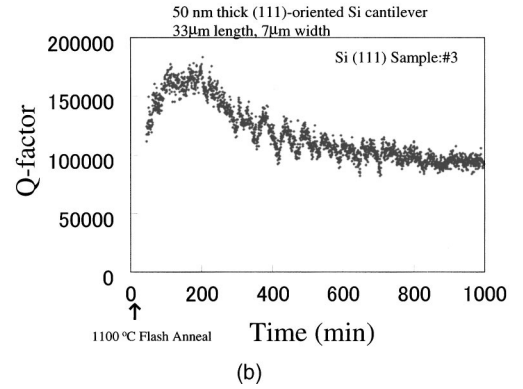


FIG. 3. (a) Resonant frequency and (b) Q factor of Si(111) cantilever (sample No. 3) after heating.

This difference in the frequency behavior of (100) and (111)-oriented cantilevers originates from surface stress induced by oxygen adsorbate. The adsorption of oxygen results in the breaking of Si–Si bonds and the formation of Si–O–Si bonds. Oxygen adsorption on Si(100) surface has been reported to lead to tensile stress of 0.26 N/m per monolayer of oxygen, whereas oxygen-adsorbed Si(111) surface exhibits compressive stress of −7.2 N/m per monolayer of oxygen.[9] The tensile stress induced by adsorption hardens the spring of the cantilever, whereas compressive stress softens the spring. The resulting resonant frequency f_{stress} due to surface stress σ can be expressed as:[10,11]

$$f_{\text{stress}}=f_0\sqrt{1-\sigma(60l^2/7Et^3)}, \tag{2}$$

where f_0 is the resonant frequency of the cantilever without surface stress, E is Young's modulus, l is the length of the cantilever, and t is the thickness of the cantilever. If the effect of mass loading is ignored, for instance, frequency

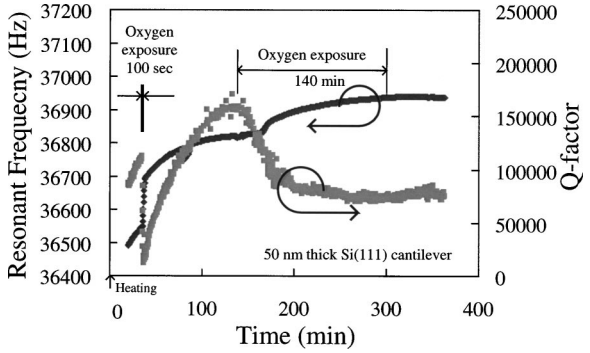


FIG. 4. Stiffening and corresponding Q factor changes by exposure to oxygen gas at a pressure of 1×10^{-5} Pa for the dots indicated in the figure.

changes of 100 Hz on Si(100) and Si(111)-oriented cantilevers (with the same dimensions as sample Nos. 1 and 3) correspond to surface stresses of 8×10^{-5} and 7×10^{-6} N/m, respectively.

If surface stress does not affect the spring constant k of the cantilever, the frequency f_{mass} after mass loading can be calculated from the adsorbed effective mass Δm:

$$f_{\mathrm{mass}}=\frac{1}{2\pi}\sqrt{\frac{k}{m^*+\Delta m}}, \tag{3}$$

where m^* is the effective mass of the cantilever. Mass loading is thus found to decrease the resonant frequency. For example, one monolayer oxygen adsorption is estimated to decrease frequencies by about 130 Hz for the Si(100) cantilever of Fig. 1 and 200 Hz for the Si(111) cantilever of Fig. 3(a). This can only qualitatively explain the behavior of resonant frequency changes depending on crystallography. In fact adsorption processes onto the silicon surface from residual gas molecules are complex phenomena, making quantitative explanations difficult.

The Q factors of both (100) and (111)-oriented Si cantilevers exhibit rapid decreases after annealing, as shown in Figs. 2 and 3(b). Residual gas molecules, including water, oxide, and hydrocarbon gradually contaminate the surface and form microscopic surface defects at dangling bonds. To investigate the influence of oxygen adsorption on the cantilever, a cantilever (sample No. 4) that was cleaned by heating was exposed to O_2 gas, and time dependencies of the resonant frequency and Q factor were measured, as shown in Fig. 4. After heating the cantilever for 35 min, oxygen gas was introduced into the measurement chamber through a precisely controlled conductance valve at a pressure of 1×10^{-5} Pa for 100 s (about 8 L). As a result, resonant frequency was found to increase and Q factor to decrease drastically. After gas supply was stopped, both frequency and Q factor tended to increase. At 137 min after heating, O_2 gas was reintroduced into the chamber for 140 min (about 620 L) at the same pressure as the first time. As can be seen, the Q factor decreases gradually in comparison to the first introduction of gas, then approaches a constant value. After several days keeping the sample in UHV, resonant frequency variations became stable within 2–3 Hz/(10 h), corresponding to 5 ppm/h.

Exposure of silicon surface to oxygen molecules is known to result in chemisorption at silicon adatom sites or at adatom back bonds of the Si(111) 7×7 surface as molecular oxygen or atomic oxygen.[12] The rapid decrease in Q factor and increase in resonant frequency are caused by this adsorption. Molecularly adsorbed oxygen at adatom sites is not stable and is gradually desorbed.[12] The increase in Q factor after first exposure to oxygen is possibly related to the desorption process.

A microscopic understanding of this effect has not been totally achieved. Dissipation of resonating structure based on phonon relaxation between two level defects has been proposed, and this theory explains the behavior of energy loss at low temperature well.[13] The thermally activated internal loss mechanism is governed by the Debye relaxation equations[14] in which temperature dependence of energy loss (1/Q) exhibits a maximum value at a low temperature corresponding to defect activation energy. Above the temperature corresponding to the activation energy, loss is expected to be inversely proportional to the square of temperature. Temperature dependence of the 50-nm-thick Si(111)-oriented beam in the range of 300–700 K was measured but the loss behavior did not fit the above mechanism in the measured temperature range.

In summary the time dependence of mechanical properties of cleaned cantilevers that were heated at 1000 °C in ultrahigh vacuum (UHV) were reported. Adsorption was found to induce stress changes in resonant frequency, with different behavior found for the different crystallographic orientations of Si(100) and Si(111). Mechanical energy loss of the resonating cantilever was found to be very sensitive to surface state. Contamination, adsorption, and the resulting defects cause energy losses on the surface, and adsorption of oxygen molecules abruptly decreases Q factor. To understand the mechanical properties, further studies, for instance the temperature dependence of cleaned samples, are needed in UHV.

This work was supported by the Japanese Ministry of Education, Science and Culture under Grant-in-Aid Nos. 13305010, 14702023, and 12131201. A part of this work was performed at Venture Business Laboratory, Tohoku University.

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